CRDEC-TR-323

LABORATORY INVESTIGATIONS PREREQUISITE
TO THE PILOT PRODUCTION
OF WET-PROCESS, GAS-AEROSOL MATERIAL

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Bernard V. Gerber
Herbert A. Hoffman

RESEARCH DIRECTORATE

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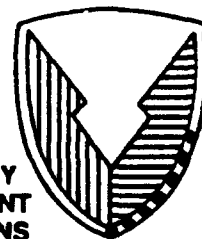
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Gerber, Bernard V., and Hoffman, Herbert A.

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
CDR, CRDEC*, ATTN: SMCCR-RS, APG, MD 21010-5423

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POC: Joseph J. Vervier, SMCCR-RS, (410) 671-3250
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13. ABSTRACT (Maximum 200 words)

Laboratory investigations are reported that resulted in a laminated and heat sealed fibrous structure formed on paper-making equipment and integrally containing charcoal and filtering glass fibers. The process methods and the formulation variables are reported.

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SUMMARY

OBJECTIVE

The objective of Subproject No. 4-80-12-007-05 was to develop improved filter materials. The objective of this work was to develop, on a laboratory scale, a material that integrally contained gas- and aerosol-filtration properties and would be capable of manufacture by standard paper-making machinery. This material would be used in various protective end items for which dry-process, gas-aerosol material was used.

RESULTS

The laboratory investigations resulted in a heat-bonded material composed of two outer water-repellent, charcoal-free layers and four or six inner core layers that contained gas-adsorbing ASC whetlerite and aerosol-filtering fine glass fibers.

Prior to heat bonding, the individual layers were formed, creped, and dried by laboratory procedures comparable to paper mill standard practice.

The proposed detailed composition of the slurry follows:

1. **Outer Layers.**

   Composition per layer follows:

   > Viscose rayon (1/4 in., 1-1/2 denier) 0.500 g/100 cm^2, 45.4%.
   > Vinyon HH fiber (3 denier) 0.500 g/100 cm^2, 45.4%.
   > Glass fiber (0.5-0.75 microns) 0.100 g/100 cm^2, 9.2%.
   > Water-repellency by silane treatment.

2. **Inner Core Layers (Four Each).**

   Composition per layer follows:

   > Viscose rayon (1/4 in., 1-1/2 denier) 0.533 g/100 cm^2, 10.2%.
   > Vinyon HH fiber (3 denier) 0.533 g/100 cm^2, 10.2%.
   > Glass fiber (2-3 microns) 0.133 g/100 cm^2, 2.56%.
   > Glass fiber (0.5-0.75 microns) 0.117 g/100 cm^2, 2.24%.
   > Charcoal furnished (80-150 mesh) 4.444 g/100 cm^2.
   > Charcoal retained (80-150 mesh) 3.900 g/100 cm^2, 74.8%.

3. **Inner Core Layers (Six Each).**

   Composition per layer follows:

   > Viscose rayon (1/4 in., 1-1/2 denier) 0.357 g/100 cm^2, 10.2%.
   > Vinyon HH fiber (3 denier) 0.357 g/100 cm^2, 10.2%.
   > Glass fiber (2-3 microns) 0.090 g/100 cm^2, 2.60%.

3

Glass fiber (0.5-0.75 microns) 0.080 g/100 cm^2, 2.30%.
Charcoal furnished (80-150 mesh) 2.957 g/100 cm^2.
Charcoal retained (80-150 mesh) 2.600 g/100 cm^2, 74.7%.

The completed, laminated, and heat-bonded materials exhibited the following average filtration characteristics in comparison with the desired specifications:

	Laboratory wet-Process	Desired Characteristics
ΔP at 320 cm/min	41-45 mm water	<50 mm water
DOP% at 320 cm/min	0.03-0.07%	0.07%
α (filtration efficiency)	7.78	6.31
Water repellency	21 in. water	20 in. water
CK life in minutes (4 mg/L 12-1/2 Lpm breather)	17-18 min	--
PS life (8 Lpm) (50 mg/L)	16-18 min	--

As determined from the current user tests, these materials exhibited adequate physical properties such as flexibility, toughness, and tensile strength to permit fabrication into the filter elements used in the E13 mask and subsequent rough handling of the entire mask.

CONCLUSIONS

A material was prepared in the laboratory and satisfied the following objectives:

1. That the material integrally contain gas- and aerosol-protective properties equal or superior to the E17 dry-form material.

2. That the material exhibit physical properties sufficient for use as a replacement for the E17 dry-form material in the current end items such as the E13 gas mask.

3. That the laboratory wet process for manufacturing gas-aerosol material be potentially capable of being scaled up to mill production on readily available commercial paper-making facilities.

PREFACE

The work described by the authors[*] of this report was authorized under Subproject No. 4-80-12-007-05, Filter Materials. This work was started in March 1956 and completed in October 1956.[**] The experimental data are recorded in laboratory notebook 4618.

The following report was recently discovered in the files at the U.S. Army Chemical Research, Development and Engineering Center (CRDEC)[**] and summarizes work, which made a major contribution to the successful fielding of the M17 gas mask. Because of its historical importance, this work is being documented and made available for future researchers. Old nomenclatures and project numbers have been retained for historical purposes and may not reflect the latest guidelines for publications within CRDEC. However, in this instance, historical accuracy is more important than current format.

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[*]Bernard V. Gerber retired from CRDEC in January 1985. He presently resides in Havre de Grace, MD. Herbert A. Hoffman was a Scientific Enlisted (military) assistant whose present location is unknown.

[**]At the time this study was conducted and completed, CRDEC was known as the U.S. Army Chemical Warfare Laboratories (CWL).

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I. INTRODUCTION

A. Objective.

The objective of this work was to develop, on a laboratory scale, a material, which integrally contains gas and aerosol filtration properties and is capable of manufacture by standard paper-making machinery. This material could be used in various protective end items for which the more costly dry-process, gas-aerosol material is presently used.

B. Authority.

This work was performed under Subproject No. 4-80-12-007-05, Filter Materials, for the fiscal years 1956 and 1957.

II. HISTORICAL BACKGROUND

Before 1950,[*] individual respirators for chemical, radiological, and biological air purification were designed around a canister containing a separate aerosol filtration layer and gas-adsorption bed. The general purpose gas-adsorption bed, in all contemporary canister designs, is composed of ASC charcoal. Charcoal improvement was directed by investigations of the base material, methods of activation, and the impregnating solutions.

The historical evolution of aerosol filter materials from Type 1 through Type 8 is covered in CRLR 291.[1]

The concept of a combined gas and aerosol filter material wherein the aerosol-filtering fibers and the charcoal are intimately mixed was first reported by Jonas.[2] The result of this work was a machine-made material, essentially Type 6 paper, with N-182 activated charcoal added to the slurry prior to formation and designated E10. The material contained a charcoal loading of 0.726 g/100 cm^2, had a DOP penetration of 0.073%, and a pressure drop of 140 mm of water at 850 cm/min flow. Although the charcoal loading was low, the material was promising. The recommendations were to develop material with higher charcoal loadings. It is interesting to note that activated charcoal rather than ASC charcoal was used because it was thought at the time that the impregnation, especially the chromium, would be destroyed by water. In view of the potential of a combined gas-aerosol material, a canister-less gas mask was designed and designated E52.

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[*]At the time this study was conducted and completed, the U.S. Army Chemical Research, Development and Engineering Center (CRDEC) was known as the U.S. Army Chemical Warfare Laboratory (CWL).

Due to the high pressure drop of the E10 material, a low resistance Type 5 matrix was co-formed with activated charcoal on a Fourdrinier machine and designated E11.[3] This material had a charcoal loading of 1.57 g/100 cm^2 and aerosol-filtration properties similar to the Type 5 matrix (although of higher resistance). A modification using copper impregnated charcoal (Type A) was also made and designated E11R1.[4] In all materials (E10, E11, E11R1), a gas-adsorption life comparable to that afforded by a canister could only be realized by using large areas or multilayers of material. In addition, if impregnated charcoals were affected by formation with the matrix from water, agents such as cyanogen chloride could not be efficiently removed under humid conditions.

Therefore, investigators assumed that dry-formation processes would lead to the most immediate development of all purpose gas-aerosol material. In addition, initial material development was directed toward use in a civilian mask because the gas-life requirements would be less stringent (less charcoal) than a military mask, and because mass production was a necessity for this item, a combined material might very well be ideal for mass production techniques.

Because industrial capabilities for dry-formed, non-woven, and bonded fabrics were small and the field relatively new, two research contracts were concurrently obtained: (a) Contract DA18-108-Cml-1367 with the Kimberly-Clark Corporation[5] and (b) Contract DA18-108-Cml-1416 with Pittsburgh Coke and Chemical Company.[6] Both research efforts evolved the same approach to the problem with respect to general formulation and the process itself. The formulation consisted of long natural or synthetic fibers for a matrix, thermoplastic resins or fibers for bonding, pulverized ASC charcoal for gas adsorption, and chrysotile asbestos for aerosol filtration. The components were mechanically dispersed in air and deposited on a screen. Thin layers of material were made, laminated, and heat sealed together. The surface layers were free of charcoal. Kimberly-Clark started immediately with a small continuous process of original design; whereas, Pittsburgh Coke and Chemical built a laboratory noncontinuous air former to simulate the newly developed Rando-Webber continuous air former (Curlator Corporation). Kimberly-Clark investigated the use of cotton and viscose fibers in precarded form; charcoal problems involving size distribution and methods of addition; the use of vinyon HH fibers; the use of vinyl resins and Naval Stores resins for strength and charcoal geometric stabilization; the use of chrysotile asbestos; heat-sealing problems; and some cover layer (liner) problems (although Type 5 paper was mostly used for the cover layers). The material developed contained 8 to 9 g of ASC 50-150 mesh charcoal per 100 cm^2. No extensive tests were performed on the material. Pittsburgh Coke and Chemical studied the dry-form process and raw material possibilities in great detail. The company covered methods of carbon bonding, grinding of charcoal, optimum charcoal size, theoretical aerosol-filtration considerations, and the use of synthetic fibers. After determining experimentally that the Rando-Webber and their laboratory machine gave analogous materials, a pilot plant was designed around using the Rando-Webber and the Rando-feeder (a precarding machine). Heat sealing by drawing heated air through the material was proposed. Work was done on liners and methods for imparting water repellency to the material.

Both companies continued their investigations under additional contracts: (a) Kimberly-Clark under Contract DA18-108-Cml-2884[7] and (b) Pittsburgh Coke and Chemical under Contracts DA18-108-Cml-3863 and DA18-108-Cml-3865.[8] Kimberly-Clark was tasked to improve their existing material and to develop a series of basic techniques for fabrication of a face mask. An improved material was made by blending vinyon HH fibers and powder with the mix. Charcoal content was increased to 12-13 g/100 cm^2, and asbestos was replaced with glass fibers as proven by the aerosol filter material work.[1] A thermoplastic, water-repellent material was developed for the cover sheet.

After considering the possible methods of fabricating face-masks to employ the new thermoplastic, gas-aerosol material, Kimberly-Clark decided to use plastisol dipping, employing a mandrel. A continuous operation was designed.

Pittsburgh Coke and Chemical Company investigated the improvement of their version of a dry-formed material and also designed and built a pilot-scale plant to produce the material. Using vinyon HH and glass, improvements were made to the material. Investigators attempted to make the material more uniform but were not wholly successful. Water-repellent liners were made, and 10 in. of repellency was obtained. The pilot plant was not fully continuous as originally conceived due to mechanical difficulties.

Kimberly-Clark concluded their work on dry-form, gas-aerosol material under Contract DA18-108-Cml-4078.[9] However, the final material was not judged wholly satisfactory, and the Pittsburgh Coke and Chemical process was chosen for further development.

Pittsburgh Coke and Chemical continued work under Contract No. DA18-108-Cml-5472.[10] Their objectives were to improve the material and the pilot process. Improvements were made in all areas, including material characteristics and a continuous process. Materials could be made with varying charcoal loadings to satisfy the specifications of the proposed protective items, including a civilian mask, an infant protector, a headwound mask, and a military mask. Additional improvements were desired, especially in material uniformity, and were investigated under Contracts DA18-108-Cml-5784 and DA18-108-Cml-5785.

The idea of using micronized ASC wherlerite in a wet-formed process was reopened by Conlisk and Bulloch.[11] They found that the chromium part of the impregnation did not leach off the charcoal in water to any significant degree although the water of the suspension was colored. Accordingly, work by the U.S. Army Chemical Center and contractors was initiated to develop a wet- formed, gas-aerosol material.

III. BACKGROUND AND ANALYSIS OF THE PROBLEM

Investigations on wet-formed, gas-aerosol material were conducted at the U.S. Army Chemical Center and A.D. Little, Incorporated under Contract DA18-108-Cml-5747. The original approach was an attempt to develop a one-step, integrally formed, material having a charcoal-free surface and, if

possible, to eliminate the heat-sealing step. Investigators realized that the material was dependent on the type of paper machine and process used. The necessity for charcoal-free surfaces required that at least three layers be nearly simultaneously formed and laminated while wet. A three-headbox, Fourdrinier machine was conceived to accomplish this aim, and a model was built in the A.D. Little laboratory. The process proved difficult but workable on the basis of the model. In addition, A.D. Little modified a handsheet mold to do the same thing. The advantage of this method was that the boundaries between the charcoal-free layers and the charcoal-containing layers were co-formed, and excellent adhesion resulted without predictable delamination. At first, no industrial machine was found that could be reasonably modified for the purpose. However, a new industrial paper machine called a "Rotoformer," which could accomplish this perfect boundary mixing between one liner and the core, was discovered at the Blandy Paper Company. The other liner would have to be formed by a cylinder machine in tandem and applied wet to the Rotoformer product at a later stage.

The main disadvantage to materials produced in the laboratory, but analogous to those probably produced by the Rotoformer, was their tendency to crack when bent sharply. This cracking was due primarily to the necessarily short paper fibers used in the furnish and also the relatively low internal cohesion. However, the potential uniformity of the wet-process material, the great production capability, and the improvement to gas and aerosol filtration due to uniformity made the wet-process method quite promising. Accordingly, much work was attempted to improve the wet-process material by the inclusion of binders. This work was partially successful, and improvements were made although the material did not approach dry-formed material in physical properties. Laboratory results indicated that the point of diminishing returns had been reached with this formulation and process, and that a redesign of the military mask (E13) to protect such material (encapsulation) would be necessary if the wet- instead of the dry-form process was to be used. Other wet processes, also necessarily using short fibers, would require the same protection; for this reason, the type of paper-making machine (originally the basis of the approach to the problem) would be unimportant.

Although the inherent filtration advantages of Rotoformer material may have supported a decision to alter the mask design to accommodate such material, deliberation on this decision was held in abeyance because an apparently feasible way of making a creped, laminated, and heat-bonded structure was conceived at CWL. The preliminary results are reported by Condon.[12] These results were so promising that A.D. Little was instructed to halt work on the Rotoformer at Blandy Paper Company and concentrate on the CWL process.

The process of crepeing paper consists of compressing the wet sheet along its length by suitable means so that small pleats are formed and dried in place. Such paper can be stretched considerably without rupture. If sheets of creped paper are laminated and bonded together at points to make a thick material, considerable flexibility is imparted because a necessary condition for bending a thick material is that the outmost layer stretch to accommodate the bend. Therefore, the disadvantages of short fibers in the formulation are overcome.

The process would consist of forming, crepeing, and drying individual layers, with and without charcoal, on a Fourdrinier machine and laminating and bonding the structure later.

The following experimental section covers the work done at CWL on the associated problems of developing this material to meet the requirements presently determined for the E13 military mask. A.D. Little had the responsibility for developing the mill process for the wet-process material.

IV. EXPERIMENTATION

A. Mechanical Design of Laminated Structure.

1. Feasibility of Crepeing in the Laboratory.

a. Procedure and Equipment.

In Section III, the advantages of using creped layers as components of the final gas-aerosol material structure are discussed. Prior to imparting crepe, the sheets were formed as they were in all subsequent experiments described in this report, on a Noble and Woods sheet-forming apparatus hereinafter described as the sheet mold or mold. The slurry furnish, with or without charcoal, was mixed in a Waring Blendor for 2 min at full speed prior to its entry into the mold. This sheet-making technique is the accepted laboratory procedure comparable to general paper mill operation.[13] The crepe was accomplished by passing the wet sheet, after its formation and blotting, through a vertically aligned pair of highly polished stainless steel rolls and removing the sheet from the top roll by use of a doctor blade, which also effects the crinkling. The sheet, before being creped, was blotted to varying degrees, and the percentage of solids was measured. The doctor blade consisted of a thin 12 in. by 4 in. strip of metal attached to a wooden base in such a manner that when the base was placed on a platform before the rolls, the blade was flush against the top roll and could be moved at various angles to it. The sheet was allowed to pass completely through the nip of the rolls before the doctor blade was set in place. The creped sheets were dried in an oven at 105 °C initially, and also under different conditions later. The procedure and equipment are shown in Figure 1.

b. Materials.

The technique for making crepe was first attempted on charcoal-free sheets having the following furnish:

● 1 g sulphite, a chemical wood fiber having a Canadian Standard Freeness of 600 cm².

● 1 g vinyon HH fiber, received in staple form and beaten in Valley beater at 1% consistency until dispersed. At first, difficulty was experienced in dispersing this fiber; however, a study of the problem resulted in a procedure that proved to be quite satisfactory. The vinyon HH was beaten with full beater weight, having previously added 0.5% Daxad 27 (based on fiber weight), an anionic dispersing agent.

● 1 g viscose rayon, received as 1-1/2-denier filament strands and cut to a length of 1/4 in. on a Taylor Style cutter.

● Sheets containing charcoal were next investigated for crepeing properties; these sheets' furnish is given below:

●● 9.2 g charcoal, an ASC type, 170-230 mesh, from Pittsburgh Coke and Chemical

●● 0.6 g Code 106 glass fiber

●● 0.8 g sulphite

●● 0.8 g vinyon HH

●● 0.8 g viscose

The furnish was then varied so that the effect of sheet thickness on the characteristics of the crepe could be noted.
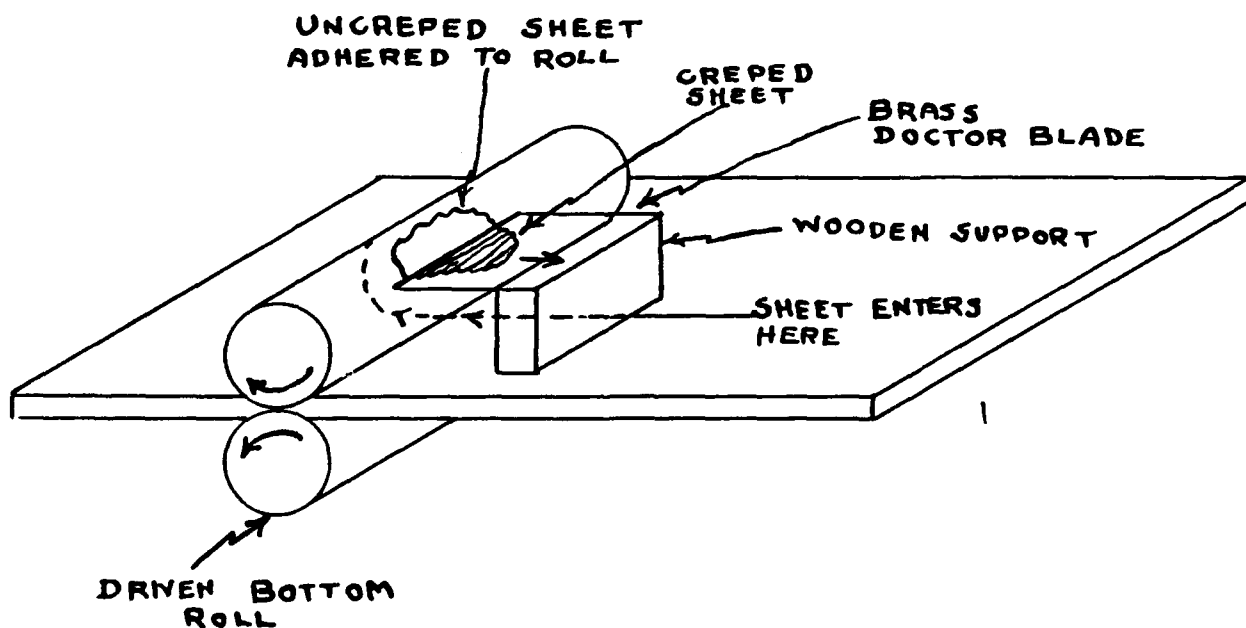

Figure 1. Procedure and Equipment Used for Crepeing

c. <u>Results and Discussion</u>.

(1) <u>Effectiveness of Method</u>.

Initial sheets received a considerable degree of crepe, amounting to about five to six furrows per inch. The degree of crepe was affected by the angle of the doctor blade, being greatest when the blade was perpendicular to the roll at the point of contact. About 85% of the sheet was creped because the last inch or so did not adhere to the roll sufficiently and could not receive the proper action. All sheets were given a definite crepe.

(2) <u>Effect of Thickness</u>.

The degree and nature of the crepe were greatly affected by sheet thickness. Charcoal-free sheets weighing 2.8 g and having a thickness of 20-25 mils received a crepe with very uniform furrows numbering six to eight per inch. Sheets containing charcoal, weighing 12 g, and 65-70 mils thick, were more difficult to crepe. The number of furrows was two to three per inch. In general, the extent and uniformity of the crepe was greatest with the thinner sheets.

(3) <u>Effect of Percent Solids of Sheets</u>.

The percent solids of the wet sheets determined the degree to which the sheets adhered to the rolls. If they were too wet, the sheets ruptured between the rolls, or the sample adhered to the bottom roll and could not be creped. If they were too dry, the sheets did not adhere sufficiently to the top roll to receive the necessary action from the doctor blade. Instead, the sheets passed over the blade missing the point of contact of blade and roll. In general, a solids content of 25-27% was necessary for a satisfactory degree of crepe.

(4) <u>Effect of Charcoal</u>.

Because the charcoal addition affected the thickness and roll-adhesion properties of the sheet, in obtaining crepe, the moisture content of these sheets was even more critical than that of the charcoal-free sheets. If the solids content was kept within the 25-27% limits, no difficulty was experienced. The charcoal was neither forced out of the sheet as a result of the blade's contact nor did a considerable amount adhere to the rolls after the sheet had passed.

2. <u>Laboratory Heat Sealing and Preliminary Evaluation of the Completed Structure</u>.

a. <u>Procedure and Equipment</u>.

The creped core layers and liners were heat sealed together to form the completed structure by passing hot air through their broad sides. Two stainless steel beakers (5 in. diameter, 6 in. length) placed top to top formed the hot-air chamber. Each beaker was fitted with a wire screen to support the sample. The upper beaker was equipped with a thermometer and side connection

leading to a cooling coil that in turn was attached to a vacuum pump. The thermometer was inserted so that its bulb was about 1/4 in. from the sample. The lower beaker was fitted with a right angled elbow projecting from the side, which was inserted into a circulating air oven at 130 °C. With the pump operating, hot air was drawn from the oven through the sample and then through the coil where this air was cooled before being discharged by the pump. Side flow was prevented by wrapping tape around the beaker butt joint. The sample was allowed to reach about 130 °C and was kept at this temperature for 10 min. The heat-sealing chamber was then removed from the heat source, and cool air was drawn through the system until the sample reached room temperature. This action completed the bond. The tape was removed, and the sample was removed for testing. The heat-sealing apparatus is shown in Figure 2.

b. Materials.

The sample to be heat sealed was composed of four creped layers and two liners, the furnish for which was given under Section A.1.b. The layers were cut into circles of 5 in. diameter and arranged so that the furrows were parallel. Lubricated glass scrim was placed on the outer sides to prevent the vinyon HH from sticking to the supporting units. The entire sandwich was then placed between perforated flexible metal plates for additional support. The structure of the material is shown in Figure 3.

c. Preliminary Test Procedures.

The completed, heat-sealed pad was evaluated primarily for general appearance, toughness, and flexibility. The pad was bent from edge to edge, ruffled, and vigorously twisted. The adhesion of the layer was noted during this rough-handling test. Prior to being tested in this manner, tests were made for pressure drop at 320 cm/min air flow.

d. Results and Discussion.

The heat-sealed pad did not delaminate when subjected to the rough-handling and bending tests. Some portions of the pad near the outer edges revealed signs of partial delamination; however, the structure in general remained intact. As expected, the pad bent more easily in the direction of the crepes than across them. The liners were sufficiently creped to stretch without rupture and contract without considerable buckling. The pressure drop of the pad was 217 mm of water, which was more than four times the desired value. The appearance of the structure and its rough-handling properties definitely warranted the studies subsequently described. The investigators believe that the formulation was responsible for the high resistance.
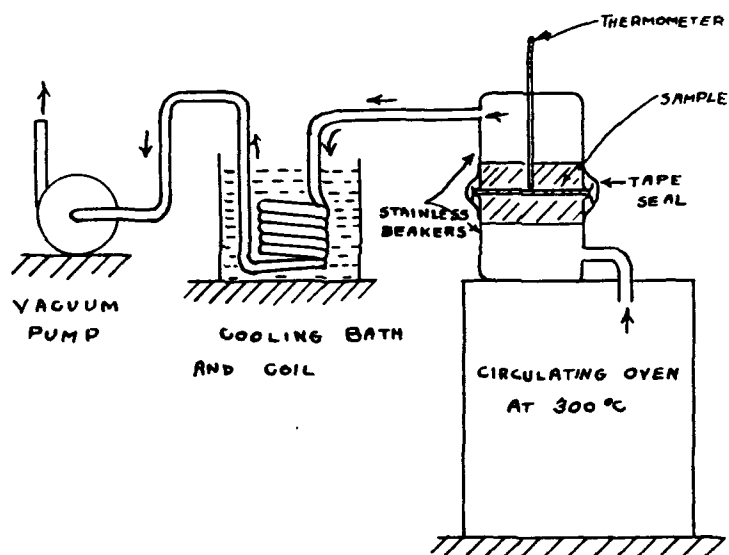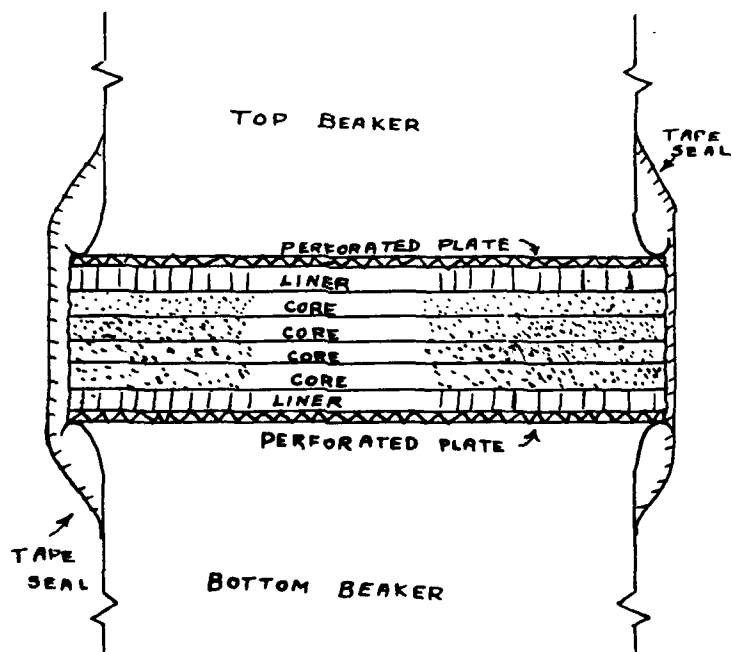
Figure 2.   Laboratory Heat Sealer

Figure 3.   Lamination of Sample for Sealing

B. Formulation.

1. General Furnish.

Initially, investigators decided to make material with approximately 12 g of charcoal per 100 cm^2 of area. At this time, from previous work, 170-230 mesh ASC charcoal was most available. A ratio of 76% charcoal to 25% fibers was selected in view of the past work done on the dry-process method. From previous work on aerosol filter materials, 20% (based on fiber weight only) of Code 106 (0.5-0.75 micron diameter) was selected as a starting point. The use of vinyon HH, viscose, and sulfite fibers, and vinyon HH powder was investigated for the cores and the liners. The area of the sheet mold was 300 cm^2. A material containing four core layers, in addition to two liners, was also arbitrarily chosen as an immediate objective.

2. Aerosol Filtration Properties.

a. Initial Results on Air Resistance.

The current aerosol filtration specifications for gas- aerosol material, which were <50 mm of water air flow resistance, <0.05% DOP (0.3 micron diameter) penetration, and measured at 320 cm/min air flow, were followed.

As mentioned, the first heat-sealed structure exhibited fine rough-handling properties but a very high air resistance. Results of the first four pads made are shown in Table 1. In view of the high pressure drop of Pad No. 1, the sulfite that was suspected of contributing excessive resistance was removed in Pad No. 2. The investigators believe that the vinyon HH, by means of possible film formation during sealing, could impart high pressure drop. Therefore, this fiber was removed from the furnish of Pad Nos. 3 and 4. Vinyon HH powder was sprinkled between layers of Pad No. 4. However, the information provided in Table 1 shows that the resistance was very high in all cases.

b. Effect of Composition of Matrix.

The authors believe that the high pressure drop of the pads could be attributable to the Code 106 fibers in addition to the matrix composition. Therefore, studies were conducted on core layers with systematically varied matrix compositions and of lower Code 106 content. Only 0.2 g of this fiber was furnished per sheet; the remainder of each sheet had 0.5 g of Code 112 substituted for the Code 106. The results of this substitution are shown in Table 2.

The data obtained showed that sulphite fiber imparts excessive resistance, at least more than either viscose or vinyon HH alone or in combination. The viscose and vinyon HH had similar independent effects on filtration as shown by Sheet Nos. 5 and 6, which had a pressure drop of only 3 mms of water.

Table 1. Furnish and Resistance of Initial Pads Composed
of Four Creped Core Layers and Two Creped Liners

Pad	1	2	3	4
Component (core layer) g				
Charcoal (170-230 mesh)	9.2	9.2	9.2	9.2
Glass fiber (Code 106)	0.6	0.6	0.6	0.6
Sulphite	0.8	-	1.2	1.2
Vinyon HH	0.8	0.8	-	-
Viscose	0.8	0.8	1.2	1.2
Component (Liner) g				
Sulphite	1.0	1.0	1.5	1.5
Vinyon HH	1.0	1.0	-	-
Viscose	1.0	1.0	1.5	1.5
Vinyon HH	No	No	No	Yes
Heat-seal treatment	Yes	Yes	No	Yes
Pressure drop	217	245	270	250

Table 2. Effect of Matrix Composition on Filtration Properties of Core Layers

Sheet	1	2	3	4	5	6	7
Charcoal (170-230 mesh)	9.2	9.2	9.2	9.2	9.2	9.2	9.2
Code 106	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Code 112	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Viscose	0.8	-	1.2	1.2	2.4	-	-
Vinyon HH	0.8	1.2	-	1.2	-	2.4	-
Sulphite	0.8	1.2	1.2	-	-	-	2.4
ΔP at 320 cm/min	9	14	12	3	3	3	38
DOP% at 320 cm/min	28	35	35	50	47	45	11

c. **Effect of Glass Fibers**.

Because sulphite fibers imparted high pressure drop, the matrix
was limited to viscose and vinyon HH in a study of the effect of glass fibers.
The Code 106/Code 112 ratio was varied with the total weight kept constant at
0.7 g. Resistance and penetration values were measured, and the results are
shown in Table 3 and Figure 4.

The pressure drop increased linearly with the weight of
Code 106 while the penetration decreased linearly. A completed pad was then
made from core layers of the Sheet No. 5 furnish to correlate single layer data
with the properties of a finished product.

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Table 3. Effect of Glass Fiber on Filtration Properties of Core Layers

Sheet	1	2	3	4	5
Charcoal (170-230 mesh)	9.2	9.2	9.2	9.2	9.2
Code 106	0.10	0.15	0.2	0.25	0.30
Code 112	0.60	0.55	0.50	0.45	0.40
Viscose	1.2	1.2	1.2	1.2	1.2
Vinyon HH	1.2	1.2	1.2	1.2	1.2
Sulphite	-	-	-	-	-
ΔP at 320 cm/min	3	4	4.5	5.5	5.8
DOP% at 320 cm/min	59	52	39	36	30

d. **Composition Providing Satisfactory Results.**

This pad had a pressure drop of 41 mm of water and a DOP of 0.025, well within the desired values of 50 mm of water, and 0.05%, respectively. The furnish is given below:

> Core (four each)
> charcoal   9.2 g
> Code 106   0.3 g
> Code 112   0.4 g
> Viscose   1.2 g
> Vinyon HH   1.2 g
>
> Liner (two each)
> Viscose   1.5 g
> Vinyon HH 1.5 g

3. **Additional Critical Properties.**

a. **Gas Life.**

(1) **Charcoal-Fiber Ratio.**

The gas life of the material is a function of the amount of charcoal present and is dependent upon the charcoal retention in the core layers. To assure that the sheet will contain the correct charcoal loading, precise calculations (based on a four-layer core) were made on the area of the sheets formed and the corresponding furnish of components.

The area of the sheets formed by the mold was 300 cm^2 The specifications for gas life of a military mask had concurrently changed to indicate a material containing 15.6 g/100 cm^2. This change necessitated 3.9 g/100 cm^2 per core layer because four layers were used to make one total core. Therefore, the charcoal required per core layer was three times 3.9 or
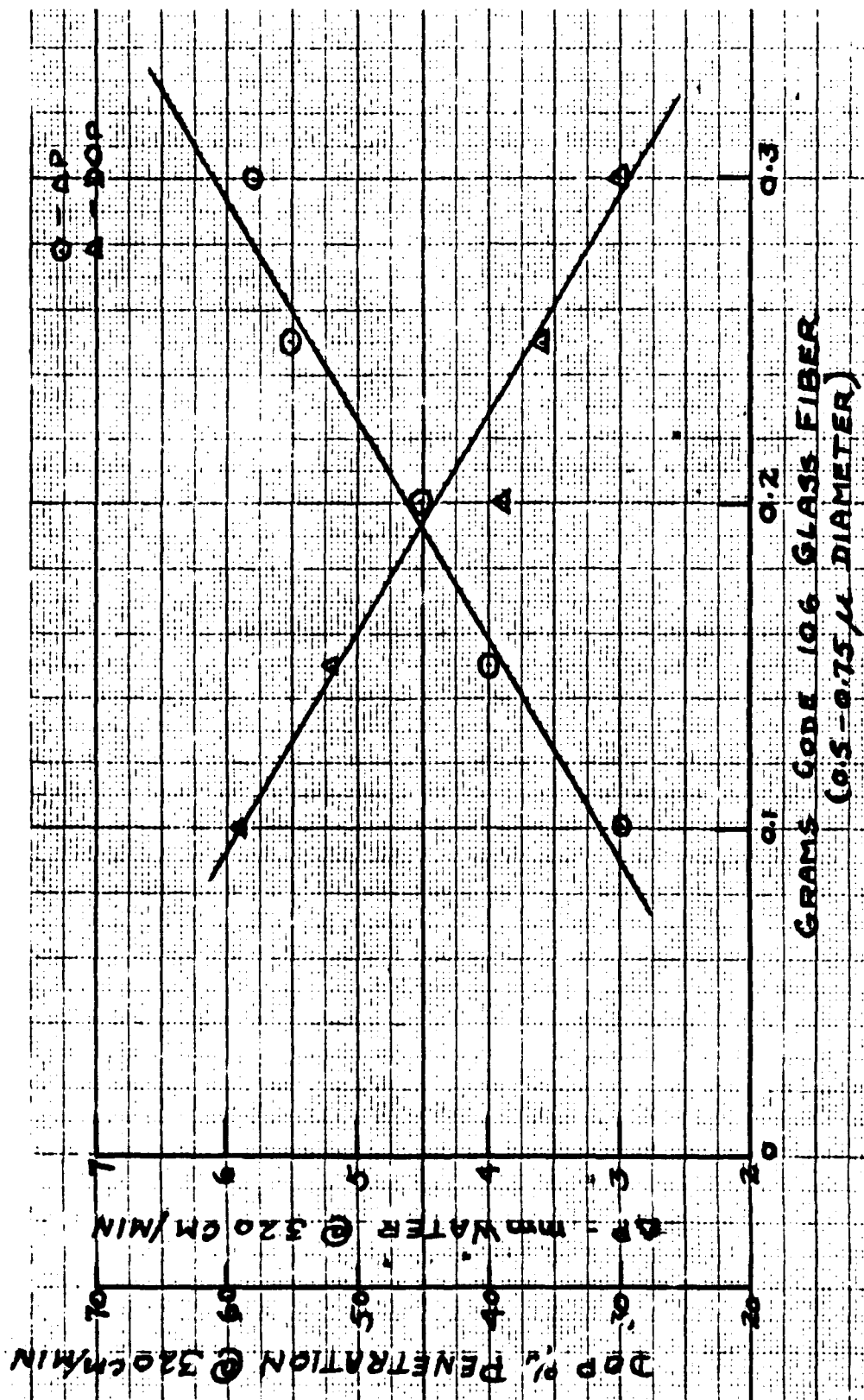
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Figure 4. Variation in DOP, % Penetration, and ΔP, Air Resistance, with Change in Glass Fiber (0.5-0.75 Micron Diameter)

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11.7 g. The specified charcoal-fiber ratio of 75/25 dictated that the fiber content of each core be 11.7/three cores or 3.9 g. Thus, the furnish, based on results of the previous data, was changed as follows:

Core layers (four each)
Charcoal   11.7 g (170-230 mesh ASC)
Code 106   0.3 g
Code 112   0.4 g
Vinyon HH   1.6 g
Viscose   1.6 g

Liners (two each)
Viscose   1.5 g
Vinyon HH   1.5 g

The glass fiber content, the most critical component from the standpoint of pressure drop and DOP penetration, was not changed. To check the filtration efficiency of this furnish, which was different from that of the pad shown previously cnly in the amounts of charcoal and matrix fiber, two completed pads were made and tested. Results were $\Delta P = 43$, DOP = 0.03% for one, and $\Delta P = 45$, DOP% = 0.05 for the second, essentially the same as the values for the previous furnish.

(2) Charcoal Retention.

To place the necessary amount of charcoal in the material and impart precision to any gas-life evaluation, a study of charcoal retention was undertaken. Sheets without charcoal, to be used as controls, were first made, dried, and weighed. Sheets with the same fiber furnish with charcoal added were then prepared and weighed. The loss in weight of the charcoal sheets minus the fiber loss incurred in the controls was taken as the charcoal loss. All sheets were weighed in an aluminum foil wrapper to prevent moisture absorption and fallacious dry weights. This technique assumed that the controls' loss in fiber was the same as the charcoal sheets' fiber loss, which for all practical purposes satisfactory for the accuracy required.

Initially, 170-230 mesh charcoal was used, and many measurements were taken of retention and gas life. However, results were confusing and showed that a defective charcoal was being used. Retention was 75%, and varying the matrix composition did not improve the value.

A series of sheets were made using different charcoal mesh size fractions from 50-200 mesh; and visual observations determined that 80 mesh charcoal was the largest size that could be used without excessive stratification on the wire side of the sheet. In addition, 150 mesh was chosen as being the smallest reasonable size from charcoal grinding data.[6] This same grinding data indicated that a 50% yield was to be expected for the 80-150 mesh fraction from roller mill treatment of 12-20 mesh charcoal.

Ten thousand pounds of grade 1 ASC were ground at Pittsburgh Coke and Chemical for the A.D. Little contract. The 50-lb sample withdrawn for CWL use had the following distribution:

Mesh	%
on 70	0.0
70-80	4.1
80-100	24.7
100-120	31.0
120-140	24.1
140-170	12.0
through 170	4.1

This charcoal was accordingly used in all subsequent work. Retention studies made showed an 88% retention of the charcoal in the furnish. To compensate for the 12% loss, additional charcoal was added.

(3) PS and CK Gas-Life Values.

Four completed pads having the known loading of 15.6 g/100 cm^2 were tested to life against PS and CK. The test results follow:

Pad	PS life min*	CK life min**
1	16	17
2	18	17
3	17	18
4	18	18

These values were considered to be satisfactory.

b. Water Repellency.

(1) Degree Required.

The liners of the finished pads needed to be resistant to water penetration to protect the inner charcoal-laden core. This water repellency or sizing should be at least sufficient to support a head of 20 in. of water.

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*8 Lpm constant flow, 50 mg/L; charcoal tested as received, test gas conditioned to 50% relative humidity (RH).

**12-1/2 Lpm breather, 1 Lpm constant flow, 4 mg/L; charcoal conditioned with air flow at 80% RH then tested with gas conditioned to 80% RH.

(2) Method of Sizing Used.

(a) Theory of Treatment.

The procedure consisted of exposing the liners to vapors of dimethyl trichlorosilane. The theory of the reaction is that when a chlorinated silane is hydrolyzed by the water adsorbed on the fibers in the sheet, a silicone film is formed over the surface of the fibers, making the sheet repellent to water. Hydrogen chloride vapor is given off during the reaction.

(b) Materials and Equipment.

In concurrent work done under Contract DA18-108-Cml-5747 at A.D. Little, liners composed of vinyon HH, viscose, and a small percentage of cotton linters did not give the necessary repellency when treated with the silane. The authors postulate that the high porosity of the sheets was responsible for this deficiency, that is, water was merely escaping through the holes in the sh t.

In this work, the standard liner (50% viscose, 50% vinyon HH) was modified with a portion of Code 106 glass to decrease the porosity and then subjected to the silane treatment. Initially, sheets were merely held over a dish of silane for a period of time during which the silane vapors rose to contact the sheet. In view of the poor results and awkwardness of technique, this method was abandoned, and a more elaborate method was adopted. The sheet was placed in a paper holder, a chamber consisting of two hemispherical iron pieces fitted with inlet and outlet tubes. One tube was connected to the side arm of a flask containing the silane, and the other tube was attached to a glass tube leading to a bubble column filled with NaOH. With the paper clamped in place, air was blown through the flask above the surface of the silane to pick up its vapors, through the paper, and then to the bubbles where the HCl was neutralized. The apparatus used is shown in Figure 5.

(3) Results and Discussion.

Results of the water-repellency testing are shown in Table 4. Runs 1 and 2 show that the water repellency was increased by 3 in. of water by using the apparatus rather than the dish method. Runs 3 and 4 show that increasing the time of a run did not improve sizing. Runs 1-6 prove that the water repellency was definitely increased by increasing the amount of Code 106 glass fiber in the liner.

For the test method used, no more than 0.3 g of Code 106 was to be added to the liners because it would detract from aerosol-filtration properties of the core. With this amount, a sizing of 16.5 in. of water was attained, and variations in procedure were tried to increase this value. Three process changes were made: (a) The silane was heated to 50 °C with a water bath to increase its vapor pressure; (b) The intake air was dried before entering the system to increase the air's capacity for carrying the

silane; and (c) NH$_3$ was subsequently passed through the system to neutralize any HCl deposited on the sheet. The sample was aged overnight before being tested and resulted in a satisfactory sizing with 21 in. of water supported.
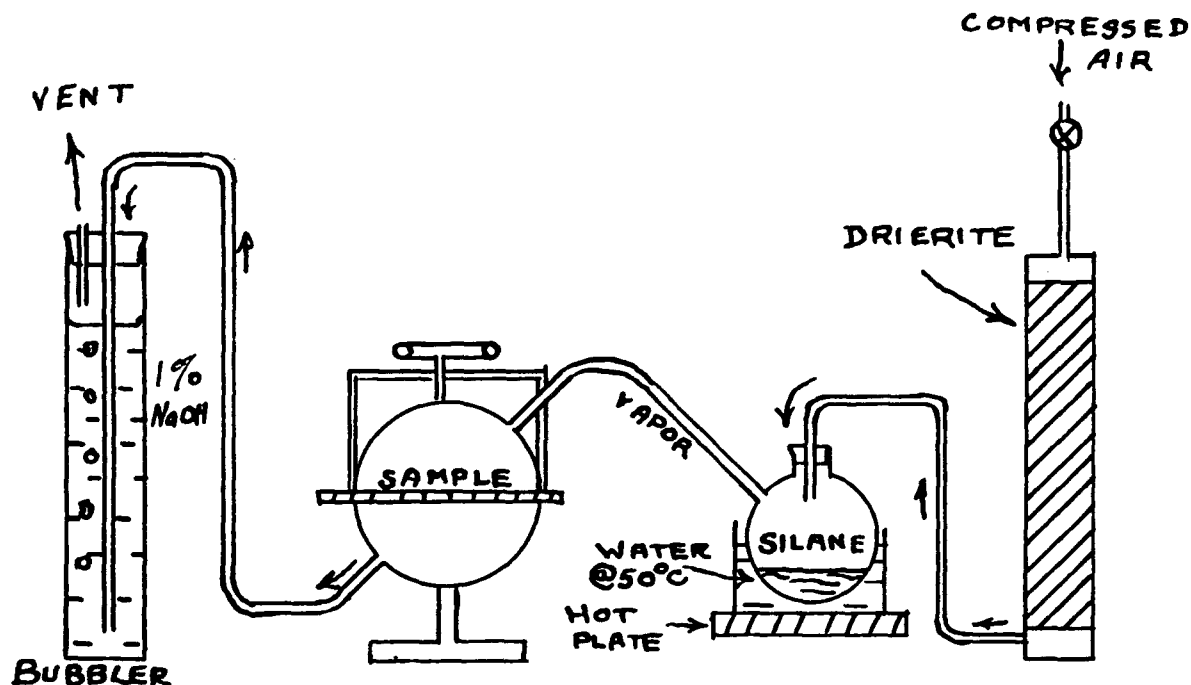
Figure 5. Apparatus for Silane Treatment

Table 4. Sizing Liners with Silane Treatment

Code 106 in liner (g)		Silane treatment	Sizing (in. of H$_2$O)
1	0.0	Sheet held over silane for 5-min	6.0
2	0.0	Apparatus, 5-min run	9.0
3	0.1	Apparatus, 5-min run	12.0
4	0.1	Apparatus, 30-min run	12.5
5	0.2	Apparatus, 10-min run	16.0
6	0.3	Apparatus, 10-min run	16.5
7	0.3	Apparatus, 10-min run, silane heated to 50 °C	18.0
8	0.3	Air dried before use, 10 min, 50 °C, NB$_4$Cl passed through; aged overnight	21.0

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4. Changes in Core Furnish to Compensate for Code 106 in Liners.

Because the total Code 106 required per pad was 1.2 g (0.3 g per core layer) for satisfactory pressure drop and DOP, and because 0.3 g is needed for each liner for sizing, the glass fiber in the core furnish was decreased to 0.6 g or 0.15 g per core layer. Pads of this furnish were prepared but had poor filtration properties, ΔP = 25 and DOP% = 0.80. Obviously, these pads were deficient in Code 106.

To determine if the liners contained less than the 0.3 g of Code 106 added, ash determinations were made; the retention was shown to be 90-95%. The relatively low glass retention rate indicated that the poor filtration properties of the pad were due to a deficiency of Code 106 fibers in the core. Therefore, core layers were prepared with increasing amounts of this fiber and tested for pressure drop and DOP. Results of the tests are shown in Table 5.

Table 5. Effect of Increasing Code 106 in Furnish on Filtration Properties of Core Layers (Charcoal Mesh, 80-150)

Core Layer	1	2	3	4	Liner
Grams Code 106 glass	0.15	0.20	0.25	0.30	0.30
ΔP, mm water	3.0	4.0	4.5	5.0	3.5
DOP%	54.0	50.0	46.5	39.5	35.5

Again, the pressure drop increased and DOP decreased with increasing amounts of Code 106 fiber. Because the liner had a lower DOP than the core layer with an equal addition of Code 106, a lower retention of this fiber in the core layer was indicated. These data should be compared with those in Table 3 when DOP was shown as a function of Code 106 in core layers with a smaller particle size (170-230 mesh) charcoal. The DOP of these layers was lower than the DOP of those containing 80-150 mesh charcoal with equal furnish of Code 106, indicating that the charcoal could account for a lower retention of the glass.

To determine the approximate furnish of glass fiber necessary for the pad, the following analysis was performed, as justified by Rodebush.[14] As mentioned, the pads furnished with 0.15 g of Code 106 per core layer and 0.3 g per liner had a DOP of 0.8%. The theoretical value was determined to be 1.04% by calculating the percentage of smoke passing through each layer of the pad. The first liner will pass 36%; the first core layer will pass 54% of 36% or 19.2%; the second core layer will pass 54% of 19.2% or 10.4%, and so on to a final value of 1.04%. This theoretical value was slightly higher than the actual value of 0.8% probably because of heat-sealing effects. Using this calculating procedure, each core layer should have a DOP of 25% to give a final pad figure of 0.05%. From Table 5, because an addition of 0.05 g of Code 106 fiber resulted in a decrease in penetration of about 5%, the amount of Code 106 that should be added to each core layer was calculated to be 0.45 g.

Four pads were then made, the first with a furnish of 0.30 g Code 106 glass fiber per layer, the second with 0.35 g, the third with 0.40 g, and the fourth with 0.45 g, these quantities being selected to consider that the actual DOP was lower than the theoretical. The results of variation in glass fiber content follow:

Pad	Code 106 per core layer	DOP%	ΔP	Efficiency (α)
1	0.30	0.09	42	7.23
2	0.35	0.054	42	7.78
3	0.40	0.018	45	8.30
4	0.45	0.012	46	8.52

Pad 2 was selected as the most desirable from the standpoint of its efficiency and low pressure drop. The CK life was rechecked on this sample and gave a value of 18 min; charcoal retention was 88%. The furnish for four core layers and two liners of a wet-process, gas-aerosol material made in these laboratories was finalized as follows:

Inner core layers (four each)	g/layer	g/100 cm^2 per layer	g/pad	g/100 cm^2 per pad	%
Viscose rayon (1/4 in. long, 1-1/2 denier)	1.60	0.533	6.40	2.133	10.20
Vinyon HH fiber (3 denier)	1.60	0.533	6.40	2.133	10.20
Glass fiber (Code 112, 2-3 μ)	0.40	0.133	1.60	0.533	2.56
Glass fiber (Code 106, 0.5-0.75 μ)	0.35	0.117	1.30	0.433	2.24
Charcoal furnished (80-150 mesh)	13.3	4.433	53.2	17.733	--
Charcoal retained (80-150 mesh)	11.7	3.900	46.8	15.600	74.80
Total	15.65	5.216	62.50	20.832	100.00
Liners (two each)					
Viscose rayon (1/4 in. long, 1-1/12 denier)	1.50	0.500	3.00	1.00	45.4
Vinyon HH fiber (3 denier)	1.50	0.500	3.00	1.00	45.4
Glass fiber (Code 106, 0.5-0.75 μ)	0.30	0.100	0.60	0.200	9.2
Total	3.30	1.100	6.60	2.200	100.0

C. Production Considerations.

1. Comparison of Laboratory Procedures with Paper Mill Practice.

The layers would be produced in the paper mill on a Fourdrinier paper machine, a rough drawing of which is shown in Figure 6. The furnish would be compounded in the beater, piped to a machine chest, then to the mixing and flow boxes of the paper machine. The sheet would be formed on the wire screen, pressed in the press rolls, dried on a section of drum driers, and reeled.

The laboratory methods used compared closely with this system. The Waring Blendor represented the beater, the sheet mold, the head box, and wire; and the blotting action represented the press rolls. The doctor blade for the crepeing operation could be placed on any convenient supporting roll after the second press section. All components of the sheets could be mixed in the beater. Because of its settling tendency, charcoal should be added in the head box for optimum mixing and retention.

The liner and the charcoal-bearing core materials would be made separately and reeled. The liner material would be made water repellent in a subsequent operation. The finished product would be made in a final separate operation by laminating the liners and the appropriate number of core layers in a heat-sealing process.

2. Necessity of Reeling Strength.

For production of the layers to be practical, the sheet would have to withstand tension as it passed from the drier cylinders to the winding roll or reel. The tensile strength should be at least 1.5 lb/in. for break-free reeling. For this reason, studies were carried out on the effect of matrix and curing condition on tensile strength.

3. Studies on Tensile Strength.

a. Effect of Vinyon HH.

Vinyon HH fiber imparts strength by fusing to a sheet when heated and forming bonds between the other fibers. During the course of this investigation, core layers and liners were tested for tensile strength; results were inconsistent and different from the values that would be expected theoretically. The authors believe that the curing condition of this fiber was responsible for the inconsistency and difference in values obtained; thus, the curing condition was investigated. The sheets used in the study were of the furnish initially used.

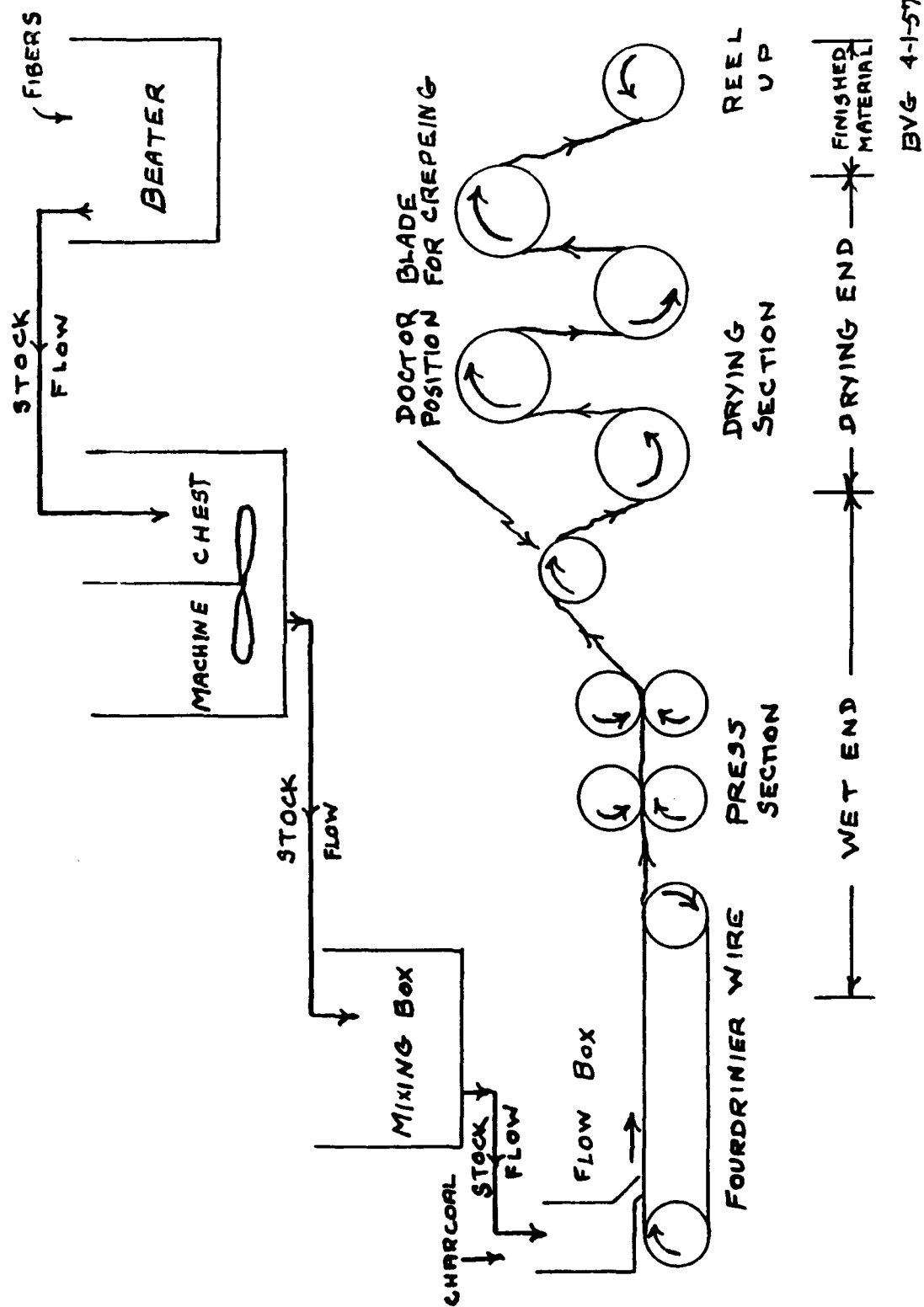| Charcoal | 9.2 g (170-230 mesh) |
Code 106	0.3 g
Code 112	0.4 g
Viscose	1.2 g
Vinyon HH	1.2 g

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Figure 6. Flow Chart for Paper-Making Process

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Samples were air dried and then cured for 30 min in an oven at various temperatures. In each case, the thermometer was placed within 1/4 in. of the sample so that a true sample temperature could be read. In addition, within the range of 70-130 °C, sheets were cured in the heat-sealing apparatus for comparison of results. These are shown in Table 6 and Figure 7 where the standard paper-testing tensile strength is shown as a function of the bonding temperature. The tensile strength reached a value of about 1.5 lb/in. at 130 °C and then tended to level off. The curve representing the curing in the heat-sealing chamber closely paralleled the oven-curing curve and represented a favorable check. It was interesting to note that the maximum tensile occurred at 130 °C, 25° over the vinyon HH softening temperature range. Core layers of the standard furnish, which have 0.4 g more vinyon HH, were cured in the oven at 130 °C and had a tensile strength of 1.6 to 1.8 lb/in.

Table 6. Effect of Curing Conditions on Tensile Strength of Initial Core Layers (all sheets dried in oven at 105 °C, before cure)

Oven Curing (30 min)	
Temperature (°C)	Tensile Strength (lb/in.)
70	0.3
90	0.4
110	0.9
130	1.5
150	1.5
170	1.8
190	1.7
Heat-Sealer Curing (30 min)	
70	0.1
90	0.3
110	0.9
130	1.5

The oven cure was actually a poor comparison to a paper machine drier cure, because the oven cure discounted the pressure placed on the sheet when it passed between the drum and supporting felt. Some sheets were dried on the laboratory drum drier at 130 °C and gave strengths of better than 2 lb/in. Therefore, it may be estimated that the tensile of sheets made in production will be at least 1.5 lb/in. and have more than the minimum reeling strength required.

b. Effect of Causticized Viscose.

Some experiments were conducted on the use of causticized viscose as a strength-imparting matrix fiber. This fiber, due to its curl and hence its ability to form strong mechanical bonds, imparted greater strength
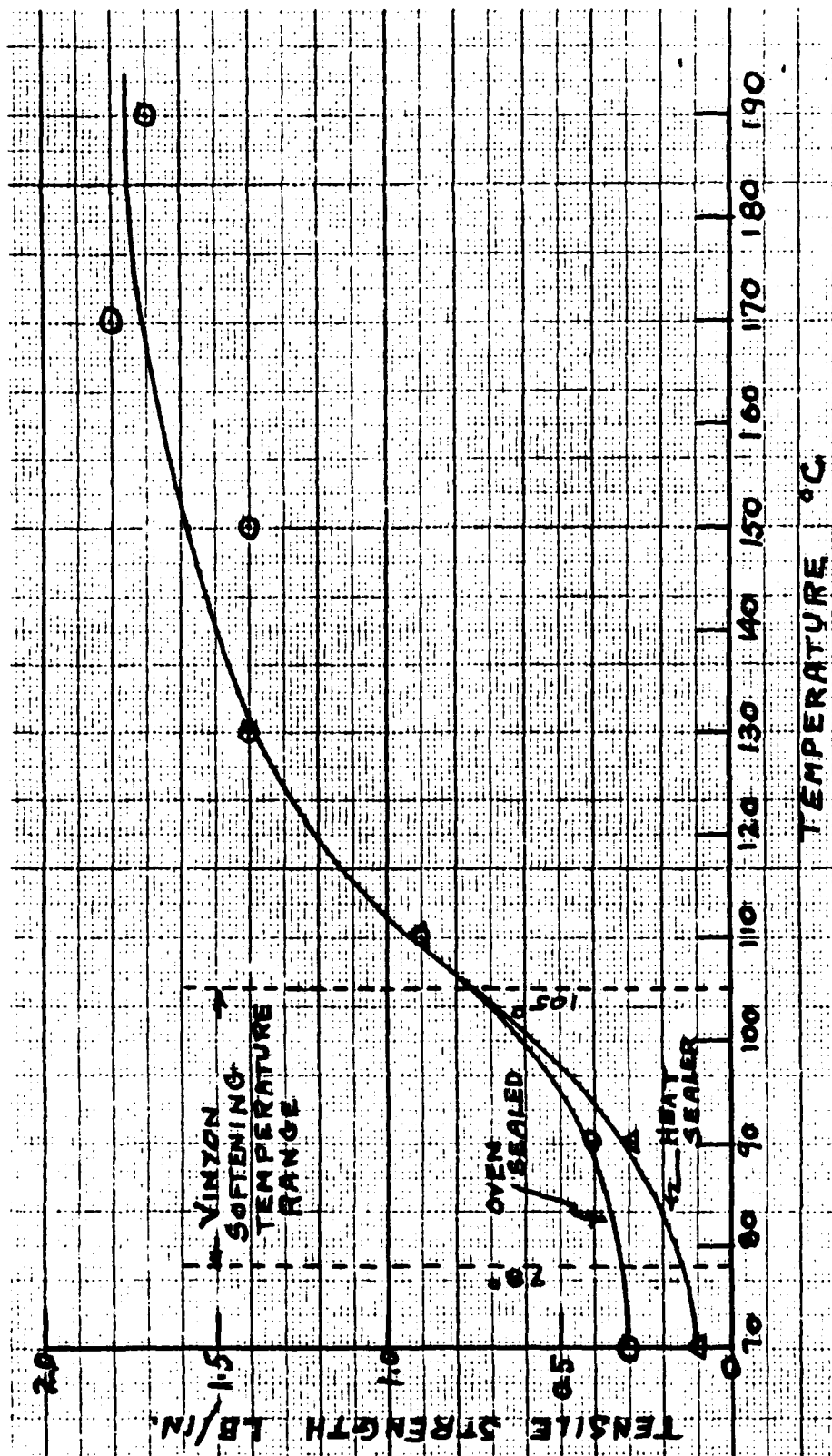
32

Figure 7. Plot of Tensile Strength of Core Layers Versus Curing Temperature

to a sheet than the unheated viscose.* However, due to the additional equipment required for mill-scale causticization, the use of this fiber in gas-aerosol, wet-process material has been deemed undesirable. Nevertheless, the use of vinyon HH fiber as a strength-imparting agent may have disadvantages (e.g., high temperature required for cure, and possibly the fusing of the sheet to the driers).

Causticized viscose was prepared in a Waring Blendor under the following conditions:[15]

Temperature	- 24 °C
Consistency	- 4%
NaOH Concentration	- 7.2%
Speed	- full
Time	- 30 sec

The fiber was quenched in cold water, washed, squeezed to a solids content of 40%, and used in the standard core layer as various substitutions for part of the vinyon HH. For example, five runs were made; the first with 1.6 g vinyon HH and no causticized viscose, the second with 1.2 g vinyon HH and 0.4 causticized viscose or 25% by weight, the third with 50% viscose, the fourth with 75% viscose, and the fifth with 100% causticized viscose. Sheets without charcoal were also made in this manner. All sheets were dried at 115 °C on the laboratory drum drier.

The effects of using causticized viscous in core layer material are shown in Table 7 and Figure 10. With the charcoal layers and controls, a synergistic effect was produced in that the curves revealed a minimum tensile at about 50% causticized viscose. In charcoal sheets, the viscose did not impart improved strength. However, the strength increased in the control sheets. Possibly, the charcoal interfered with the bonding of the curly fibers. Investigators should note that a temperature in excess of 115 °C may be necessary to develop optimum strength with causticized viscose as with vinyon HH. Data indicated that causticized viscose could be used as a vinyon HH substitute for imparting tensile strength; but, this substitution did not result in great strength improvement in the charcoal sheets.

4. Cores of Five and Six Layers.

a. Reasons for More Layers.

Depending upon the facility of producing the pads on a commercial basis, it may be desirable to build the final structure of more than four core layers. For example, if the four-ply layers are too thick to be successfully creped on the paper machine, more layers of lower caliper will be necessary.

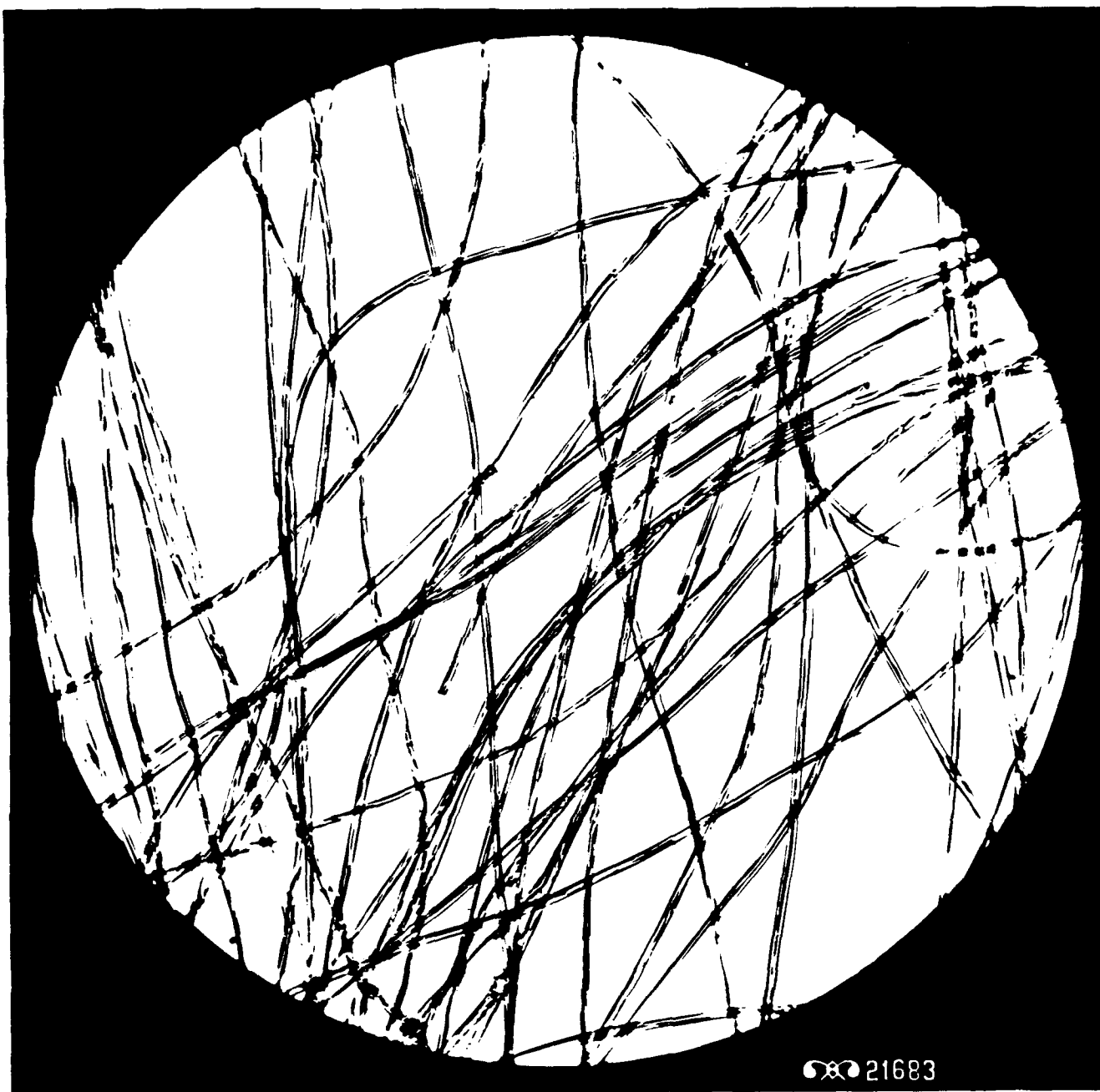*Photomicrographs of normal and causticized viscose fibers are shown in Figures 8 and 9.

Figure 8. Photomicrograph of Untreated Viscose Fibers (200x)

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Figure 9. Photomicrograph of Causticized Viscose Fibers (200x)

Table 7. Effect on Tensile Strength of Causticized Viscose
in Core Layers with and Without Charcoal

Tensile Strength (lb/in.)		Vinyon HH (g)	Causticized Viscose (g)	Causticized Viscose (based on 1.6 g total) (%)
With charcoal	Without charcoal			
1.36	1.87	1.6	0.0	0
1.15	1.26	1.2	0.4	25
0.77	1.31	0.8	0.8	50
0.73	2.05	0.4	1.2	75
1.13	2.90	0.0	1.6	100

b. **Cross and Parallel Crepeing.**

During the course of this study and the heat-sealing
experiments described previously, the authors found that cross crepeing
(i.e., arranging the layers so that the crepes or furrows of one layer are
perpendicular to those of the adjacent sheets) gave a tougher and more uniform
sheet than parallel crepeing in which the layers are arranged so that the crepes
are parallel to each other. With cross crepeing, the filter pad could bend more
easily in all directions; with the parallel arrangement, the sheet offered
resistance to bending across the crepes. For this reason, cross crepeing was
adopted as a standard practice.

c. **Five-Layer Cores.**

A few pads containing a core of five layers were made. The
charcoal retention was essentially the same as that in the previous pads. The
physical properties of these pads were poorer. The cross crepeing resulted
in the liner crepe being parallel to each other; consequently, as discussed
above, the difference in bending properties in different directions was
pronounced. Therefore, the authors decided that the core of the pad should
be composed of either four or six layers.

d. **Six-Layer Cores.**

The furnish of the layers was calculated so that the total
core composition remained the same as the pads of four-layer cores. The
retention of charcoal was again the same (88%), and the charcoal added to
each layer was calculated to give a loading of 15.6 g/100 cm^2.

The aerosol-filtration properties of this six-layer core pad
were ΔP = 39 and DOP% = 0.12. Apparently, the lighter matrix retained less Code
106 fibers. Therefore, each core layer was furnished with an additional 0.01 g
of Code 106 or 0.06 g more per pad. The ΔP and DOP%, as a result of this added
glass furnish, were 43 and 0.049, respectively, with the α value being 7.70 and
quite satisfactory. The furnish was accepted and is given below along with the
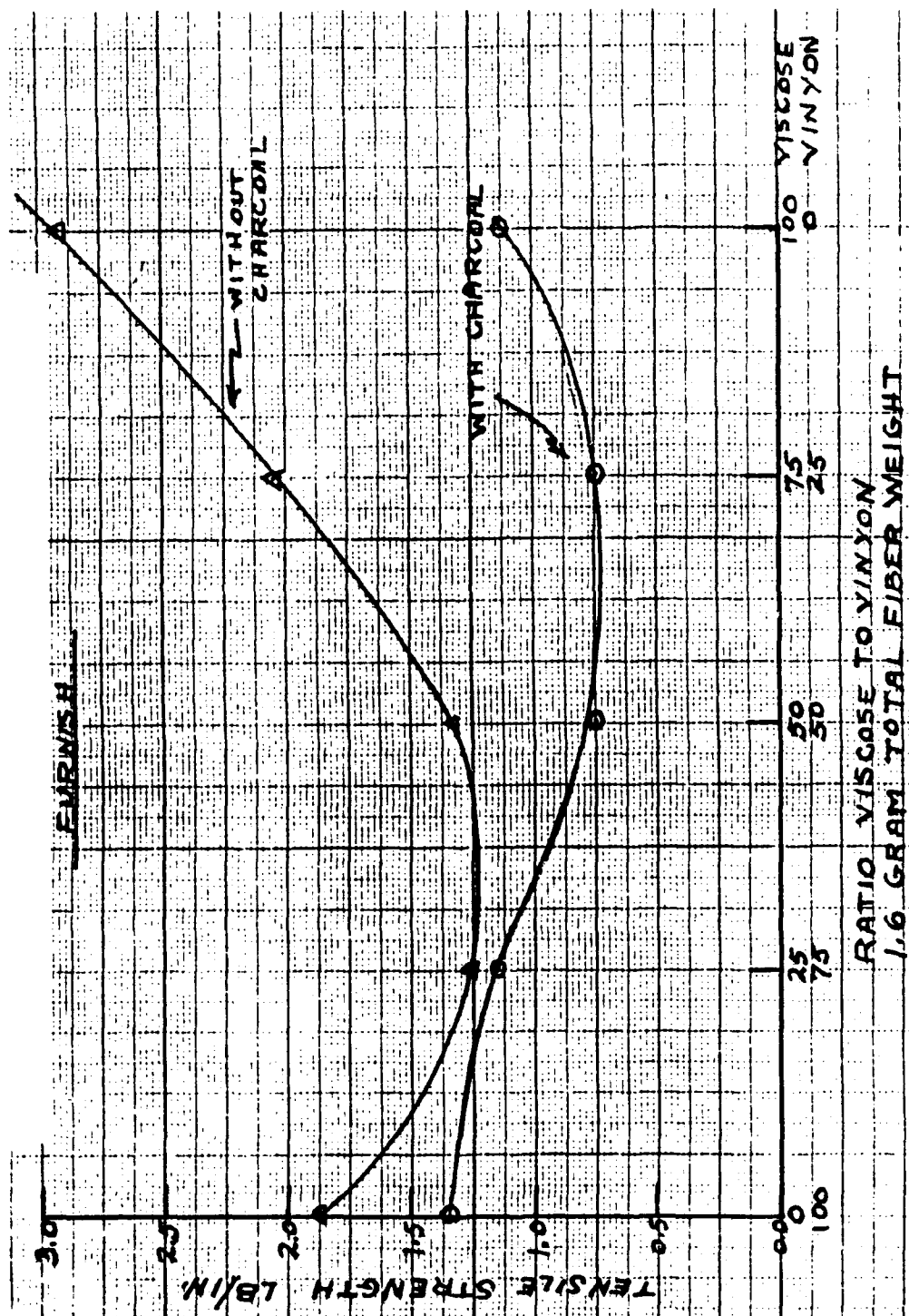four-layer and total pad furnishes.

Figure 10. Plot of Tensile Strength of Core Layer with and Without Charcoal Versus Ratio of Viscose to Vinyon HH in 1.6-g Fiber Furnish

	Per layer 4-layer formulation	Per layer 6-layer formulation	Per pad 4-layer formulation	Per pad 6-layer formulation
		Core layer		
Charcoal	11.70 (13.3)*	7.80 (8.87)*	46.8	46.8
Viscose	1.60	1.07	6.4	6.42
Vinyon HH	1.60	1.07	6.4	6.42
Code 106	0.35	0.24	1.4	1.44
Code 112	0.40	0.27	1.6	1.62
	Liner layer (two used in final structure)			
Viscose	1.5	1.5	3.0	3.0
Vinyon HH	1.5	1.5	3.0	3.0
Code 106	0.3	0.3	0.6	0.6

*The numbers in parentheses are the amounts of charcoal added; the numbers not in parentheses are the amounts of charcoal retained.

Tensile strengths of the 6-ply layers cured in the drum drier at 130 °C were measured and gave results in excess of 1.5 lb/in.

5. Tentative Specifications.

The following tentative specifications for the laboratory sheets are listed below to be used as a guide for production in the paper mill:

	4-Ply Layer	6-Ply Layer	Liner
Uncreped			
Basis weight (g/300 cm^2)	15.8	10.5	3.27
Tensile (lb/in.)	1.6-1.8	1.6	2.0
Elongation (%)	8-9	8-9	--
ΔP (mm H$_2$O, 320 cm/min)	7	3.5	3.5
DOP% (320 cm/min)	30	55	44
CK life (min, 12 Lpm)	6 (two layers)	9 (four layers)	--

	4-Ply Layer	6-Ply Layer	Liner
Creped			
Basis weight (g/272 cm^2)	15.8*	10.5*	3.25*
Solids before crepeing (%)	25-27	25-27	25-30
Tensile (lb/in.)			2.0
Along crepe	1.5	1.5	2.2
Across crepe	1.5	1.5	2.0
Elongation (%)	8-9	8-9	8-9
Extensibility (%)	10	10	10
ΔP (mm H$_2$O, 320 cm/min)	7	4	46
DOP% (320 cm/min)	30	55	46
CK life (min, 12 Lpm)	6 (two layers)	10 (four layers)	--

*The sheet area due to crepe decreased from 300 to 272 cm^2. This means that pads of creped layer would be at least as effective for filtration as pads made from flat sheets.

V. DISCUSSION

A gas-aerosol material made by standard paper-making machines had the potential advantages of manufacturing capacity and uniformity of characteristics. The paper-making process, however, is generally restricted to the use of short fibers that are capable of being dispersed in water and redeposited uniformly on a screen. The gas-aerosol material that was the goal of this project must contain approximately 16 g of charcoal per 100 cm^2. The bed depth of this weight of charcoal would in itself be approximately 1/4 in. In addition to the charcoal, sufficient fiber must be present for dimensional stability, aerosol filtration, and increased thickness. The problem of bending (without rupture) a material this thick while using short fibers is obviously difficult. Although there are several approaches possible in the development of a gas-aerosol material made by a wet process, previous and concurrent work at Arthur D. Little on a non-laminated structure showed that, although feasible, such a structure would be limited in flexibility.

The investigations reported herein show superior physical properties for a heat-bonded, laminated structure and optimum flexibility for a structure composed of individually creped layers. In this instance, the crepe undergoes the necessary extension when the material is bent without damaging the short fiber bonds. However, an acceptable material could probably be made without crepeing although it is felt that an optimum material should be creped, if at all possible.

The procedure of symmetrically laminating the creped layers at 90° to each other for more uniform physical properties was also discussed herein.

Investigations on formulation to obtain satisfactory gas and aerosol filtration were successful. Approximately 90% of the charcoal (80-150 mesh) was retained in a 75-25 ratio of charcoal to fibers, and air resistance was reasonable at the proper penetration level. An efficacious amount of vinyon HH was located for use within the charcoal-fiber ratio investigated.

Fine glass was incorporated into the liners to achieve water repellency by silane vapor treatment; however, this redistribution did not affect filtration efficiency.

Although very satisfactory material was made in the laboratory, several problems were foreseen in mill practice. For one, it was not known whether the wet web as formulated would have sufficient strength for machining. The admixture of wood fiber to gain strength was detrimental to efficiency; and although it increased the strength of the wet web, causticized viscose decreased the strength of the final product. The outcome will be dependent upon drying capacity. If the paper-machine driers can be run hot enough to partially seal the vinyon HH in the layer, a superior product will be made.

Finally, a production heat sealer must be constructed. If the material is to be cross creped, this phase will probably be a batch operation.

VI. CONCLUSIONS

A material was prepared in the laboratory and satisfied the following objectives:

A. That the material integrally contain gas- and aerosol-protective properties equal or superior to the E17 dry-form material.

B. That the material would exhibit physical properties sufficient for use as a replacement for the E17 dry-form material in the current end items (e.g., E13 gas mask).

C. That the laboratory wet process for the manufacture of gas-aerosol material would be potentially capable of being scaled up to mill production on readily available, commercial, paper-making facilities.

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GLOSSARY OF TERMS

Air resistance (ΔP)	Resistance to air flow expressed as head (pressure drop) in millimeters of water.
AR	"As received" refers to humidity condition in the PS gas-life test.
ASC	Impregnant code for charcoal; stands for silver, copper, and chromium.
Beater additive	A component added to stock prior to sheet formation.
Canadian standard freeness	The ability of a paper fiber pulp to drain free of water, measured in cm^3 of water.
CK	Cyanogen chloride.
Core	The inner layer or layers of gas-aerosol material that contains charcoal.
Crepe	A material that has been wrinkled in parallel pleats and treated to make these pleats relatively permanent.
CWL	U.S. Army Chemical Warfare Laboratory.
Denier	A textile term related to fiber diameter; the weight in grams of 9000 m of thread or filament.
Doctor blade	A metal blade held erect to a moving roller to dam up a material moving on the roller.
DOP penetration	Percent penetration of a dioctylpthalate aerosol of 0.3 micron diameter.
Furnish	Paper-makers technical jargon referring to the water suspension of the total components used to make paper. Often referred to as slurry or stock.
Furrow	Used synonymously with wrinkle, pleat, or crepe.
Filtration efficiency	$\dfrac{-100}{\Delta P} \log \dfrac{DOP}{100} = \epsilon$
Liner	The surface coverings of a laminated material, also called cover sheets.
Micron	One-millionth of a meter; equals 3.9×10^{-5} in.
mil	One-thousandth of an inch.

mold	Laboratory apparatus used to form the wet sheet of paper.
nip	The tangent point of two moving rollers.
ply	A single layer of a laminated structure.
PS	Chloropicrin.
sheet	The formed paper fibers either wet or dried. Refers to thin paper in any form.
sizing	Treating paper at any step of the operation to impart resistance to liquids.
slurry	See furnish above.
sulfite	Fibers prepared from the natural state by the sulfite process.
Vinyon HH	Copolymer of vinyl chloride and vinyl acetate.
Viscose	Synthetic cellulose fiber prepared by the Xanthate process.
water repellency	Ability to inhibit the permeation of liquid water.